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Indian Standard

CORROSION OF METALS AND ALLOYS — CORROSIVITY OF ATMOSPHERE — MEASUREMENT OF POLLUTION — METHODS

ICS 77.060

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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July 1996 **Price Group 4**

Corrosion Protection Sectional Committee, MTD 24

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Corrosion Protection Sectional Committee had been approved by the Metallurgical Engineering Division Council.

The property of an atmosphere to cause corrosion of metals and alloys is controlled by temperature-humidity complex and pollution. A basic requirement for determination of the corrosivity of atmospheres is the standardized measurement of concentration and deposition rates of environmental pollutants. The concentration of SO_2 and other pollutants in the air should be measured in accordance with special standards. The body of this standard represents methods for deposition rate measurements of SO_2 and airborne salinity.

The methods included in this standard have been choosen for their easy applicability and good comparability of results. It is important to stress that the classification of SO_2 and airborne chloride levels in IS 14191 : 1995 is based on measurement applying methods described in this standard.

In preparation of this standard assistance has been derived from ISO 9225-1992 'Corrosion of metals and alloys — Corrosivity of atmospheres — Measurement of pollution', issued by the International Organization for Standardization.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CORROSION OF METALS AND ALLOYS — CORROSIVITY OF ATMOSPHERE — MEASUREMENT OF POLLUTION — METHODS

1 SCOPE

This standard specifies three methods for measuring the deposition rates of SO_2 (sulphur compounds) and airborne salinity.

This standard does not cover concentration measurements. The concentration of SO_2 and other pollutants in the air should be measured in accordance with standard methods.

The measurement methods included in this standard apply for characterization of the test site of exposure location from the viewpoint of corrosivity. The pollution data obtained by these methods have to be used as classification criteria for the determination of corrosivity categories.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
14191 : 1995	Corrosion of metals and alloys — Classification of corrosivity of atmospheres

3 DETERMINATION OF SO_2 DEPOSITION RATE ON PbO_2 SULPHATION PLATES

3.1 Principle

Atmospheric SO_2 reacts with the lead dioxide to form lead sulphate. The plates are recovered and sulphate analysis performed on the contents to determine the extent of sulphur dioxide capture. The deposition rate of SO_2 is expressed in terms of ($mg/m^2 \cdot d$).

The lead dioxide reagent used in this method may convert other sulphur bearing compounds, such as hydrogen sulphide and mercaptans, to sulphate as well.

The inverted position of the disc is intended to minimize sulphur capture from acid precipitation or sulphuric acid aerosol.

3.2 Sampling Apparatus

3.2.1 *Sulphation Plate*

Sulphation plates may be purchased ready for exposure or may be prepared. The following method is recommended if it is desired to prepare sulphation plates.

Bond filter paper circles to the bottom of polystyrene culture (Petri) dishes. Size of 50 mm or 60 mm is convenient. The bonding process is carried out by placing a filter paper rough side up, in the bottom of the dish. The filter paper should be of a size which fits without wrinkling inside the dish. Carefully squirt, acetone into the dish so that the filter becomes just saturated. Press the paper firmly with a glass rod so that all parts of the filter are pressed into the dish. Allow acetone to evaporate.

Place a batch of bonded plates, 80 mm × 50 mm or 55 mm × 60 mm, in a rack and rinse with distilled or deionized water. Then fill the plates with water again and allow it to stand for one hour. Pour the water out and refill between one quarter to one half with distilled or deionized water.

Add 3.5 g of gum tragacanth and 900 ml distilled or deionized water to a high speed blender container. Set at a low speed and blend for two hours.

Pour the contents of the blender into a 1 litre beaker and return 350 ml of the solution to the blender container. Pulp 3.5 g of filter paper in the 350 ml of gum solution with the blender set at a moderate speed until the mixture appears smooth and uniform.

Return 400 ml of the gum solution previously removed from the blender and blend at a moderate speed for one minute.

Turn the blender to high speed and add 112 g of lead dioxide. Blend for two minutes and then turn the blender back to a low speed.

Carefully pipette 10 ml of the mixture into each 50 mm plate or 15 ml into each 60 mm plate. Make sure that the mixture spreads uniformly to the edge of each plate.

Place the rack of plates in an oven set at 40-50°C for 20 hours.

Remove the plates from the oven, allow them to cool and seal the plates with a tight-fitting cover to preserve them until the exposure begins.

Plates shall be numbered and placed for exposure within 120 days of preparation. Retain at least three plates from each batch as a reference.

3.2.2 *Exposure Rack*

Brackets shall be used to hold plates securely in inverted position so that the lead dioxide mixture faces downward. The plate shall be horizontal and shall not be obstructed from normal winds and air circulation currents. The bracket shall be fabricated from a material which has adequate corrosion resistance in the atmosphere. The bracket design shall include a retaining clip or any other provision to hold the plate in the event of strong winds. A typical bracket design is shown in Fig. 1.

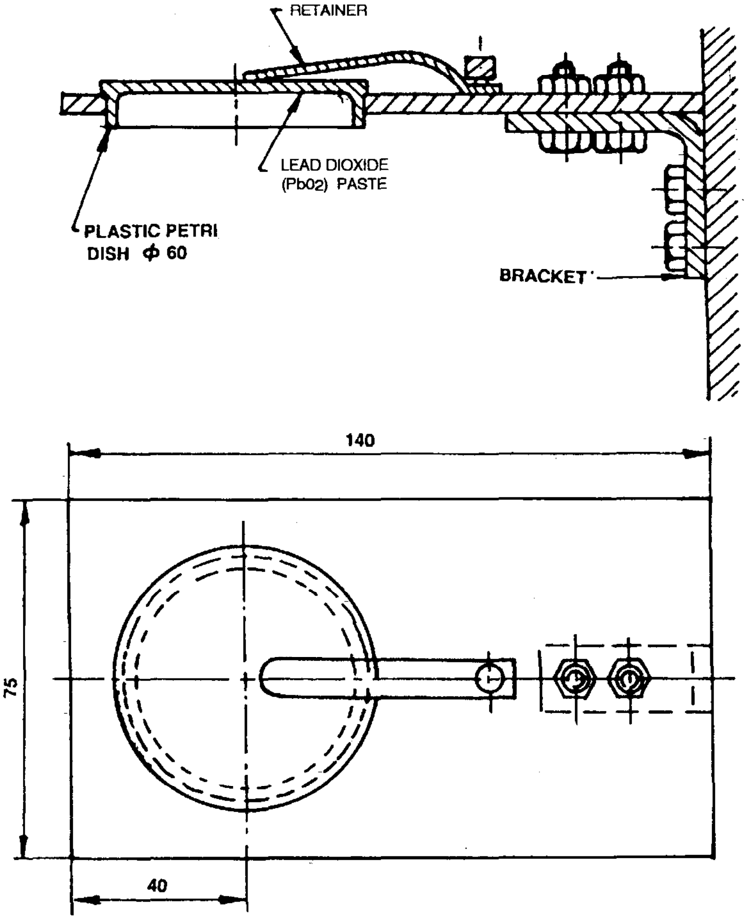
3.3 Sampling Procedure

When monitoring exposure sites, a minimum of three plates shall be used for each exposure period. As far as possible the plates should be placed, at the highest as well as lowest level above the ground the corrosion test specimens are exposed.

A 30±2 days exposure period is recommended. At the conclusion of the exposure period, the plates should be removed from the bracket and covered tightly to prevent additional sulphation. Analysis of the plates should be completed within 60 days of the completion of the exposure. The plate identification, exposure location, date of exposure initiation and completion shall be recorded when the exposure is terminated.

3.4 Sulphate Analysis

The sulphation plates shall be analysed for sulphate content using any established quantitative analysis technique. A turbidimetric method for this analysis is given in the following clauses.



All dimensions in millimetres.

FIG. 1 SULPHATION PLATE HOLDER

3.4.1 *Principle*

The contents of the sulphation plate are removed and dissolved using, for example, a solution of sodium carbonate. Thereafter, the sulphate is precipitated with the barium ion and measured turbidimetrically.

3.4.2 *Reagents*

Unless otherwise specified, during the analysis, reagents of analytical grade and reagent grade water as per IS 1070 : 1992 shall be used.

3.4.2.1 *Sodium carbonate (50 g/l)* — Dissolve 50 ± 0.5 g of anhydrous sodium carbonate ($Na_2 CO_3$) in 1 litre of water.

3.4.2.2 *Hydrochloric acid (0.7 mol/l)* — Dilute 60 ml of concentrated hydrochloric acid (r.d. = 1.19) to 1 litre with water.

3.4.2.3 *Barium chloride dehydrated solution (5 g/l)* — Dissolve 5 g of barium chloride dehydrated ($BaCl_2 . 2H_2O$) in 1 litre of water.

3.4.2.4 *Sodium sulphate* — Standard solution corresponding to 500 μg of sulphate per litre.

Weigh, to the nearest 0.1 mg, 0.740 g of anhydrous sodium sulphate (Na_2SO_4), place it in a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix well. 1 ml of this standard solution contains 500 μg of sulphate.

3.4.3 *Plate Analysis Procedure*

Quantitatively remove the lead dioxide and as much of the fibrous material as necessary from the plate into a 50-ml volumetric flask.

Add 20 ml of sodium carbonate solution and stir.

Allow it to stand for three hours with occasional stirring.

Place in a 100°C water bath for 30 minutes.

Cool and adjust volume to 50 ml.

Filter at least 15 ml through a quantitative fine grade filter paper into a clean, dry test tube. Do not wash.

Pipette 10.0 ml of filtered sample into a 150 mm long test tube of diameter 25 mm and add 10.0 ml distilled water and 5.0 ml of the hydrochloric acid solution (**3.4.2.2**). Swirl to mix and check *p*H with indicator paper. The *p*H shall be between 2.5 and 4.0. If not, discard and repeat this step.

Pipette 5.0 ml aliquot of the sample into two test tubes of 25 ml diameter and add 15 ml of water to each. Add 1 ml barium chloride solution (**3.4.2.3**), mix vigorously and allow it to stand for five minutes before measuring turbidity.

Measure the turbidity of the sample solution at 500 nm against the solution which has no barium added. Convert absorbance (turbidity) reading to micrograms of sulphate from the calibration curve obtained by the procedure below.

Blank samples consisting of unexposed sulphation plates taken from each batch of prepared and exposed plates should be analysed.

3.4.4 *Preparation of Calibration Curve*

Dilute 10.0 ml of the sodium sulphate standard solution to 100 ml using a volumetric flask.

Pipette out 1, 2, 3, 4, 5, 10 and 15 ml of the diluted sodium sulphate solution into test tubes. Dilute each to 20 ml with distilled or deionized water and then follow the procedure above.

Plot on linear graph paper, the absorbance readings of each of the above solutions against the sulphate content of the solutions, that is, 50, 100, 150, 200, 250, 500 and 750 μg.

3.5 Evaluation of Test Results

The sulphation rate is calculated in terms of SO_2 capture by the plate. The mass of sulphate obtained from the plate analysis procedure is converted to net sulphation mass by subtracting the blank value obtained from the batch of plates in question.

The deposition rate of SO_2 *R*, expressed in mg/(m^2. d), is given by the formula:

$$R = \frac{(m_1 - m_0) \times 16.67}{A \times t \times 1\,000} \quad \text{... (1)}$$

where

m_1 = content of sulphate in a plate, in micrograms;

m_0 = content of sulphates in a blank test in micrograms;

A = area of plate, in square metres; and

t = exposure time, in days.

4 DETERMINATION OF SO_2 DEPOSITION RATE ON ALKALINE SURFACE

4.1 Principle

Sulphur oxides (SOx) and other sulphur compounds of acid character are collected on the alkaline surface of porous filter plates saturated by a solution of sodium or potassium carbonate sulphur compounds which are converted into sulphates are determined. The deposition rate of SO_2

is expressed in terms of milligrams per square metre day [mg/(m^2.d)].

4.2 Sampling Apparatus

4.2.1 *Sulphation Plate*

Sulphation plates shall be prepared by the following method:

Paper filter plates of 150 mm × 100 mm × 3 mm size (it is admissible to use chromatographic paper filter plates or other materials of similar properties) shall be immersed for a period of 2 minutes into 70 g/l sodium or potassium carbonate solution.

After having been taken out and drained, they shall be dried for either 1 hour or 8 hours at a temperature of 105°C or 90°C, respectively. After drying, the plates shall be sealed into polyethylene or polypropylene plastic containers. Five pieces shall be taken off from every processed series in which the content of sulphates is determined as a blank.

4.2.2 *Exposure Rack*

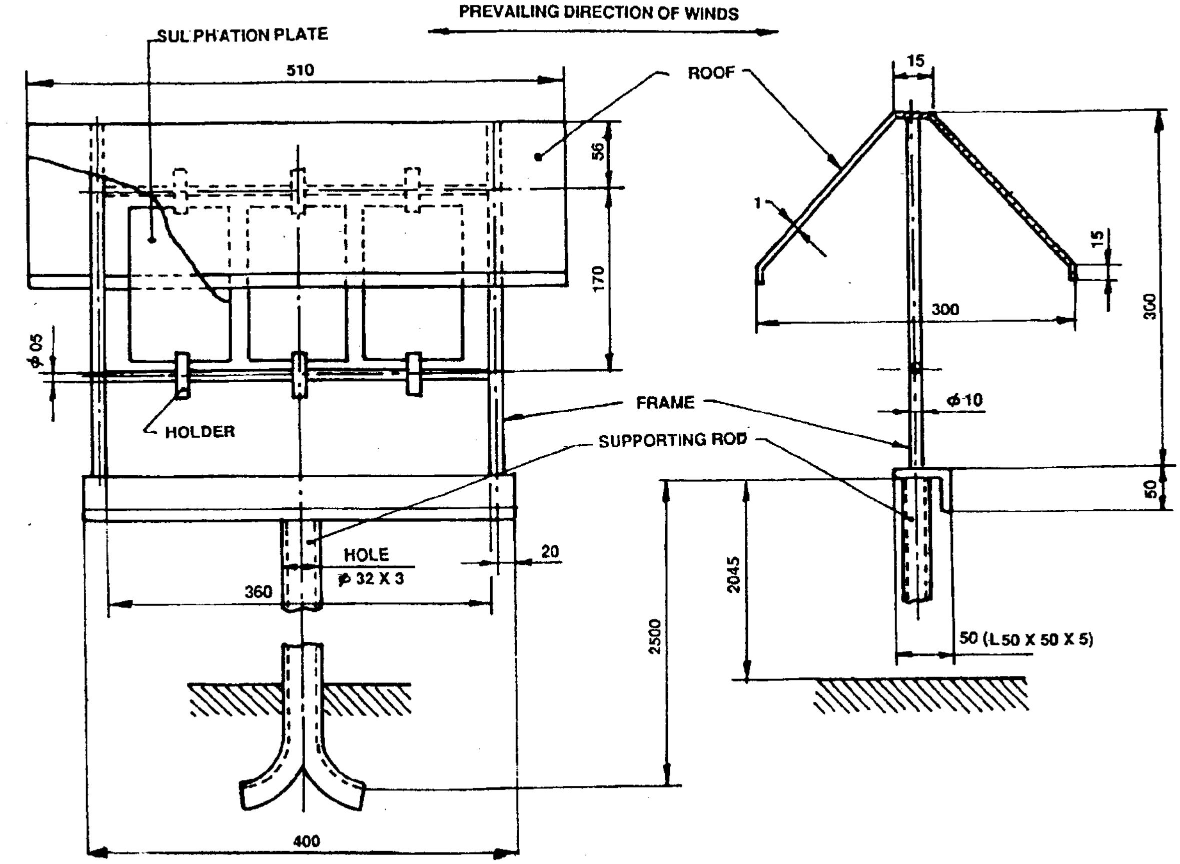
The test plates are exposed on a rack (Fig. 2) in a vertical position so that their surfaces are parallel to the prevailing direction of wind. The bottom edges of the plates shall be at a height of 1.8 m to 2 m above the ground. A roof over the rack prevents the plate washing out by rain but enables the free access of air to the plates.

4.3 Sampling Procedure

At the test location (test site) three test plates are installed by the fasteners on the rack. The time of exposure of the test plates is 30 ± 2 days unless the character of the test or the pollution level necessitates some other time (60 or 90 days). After the exposure is complete, the test plates are taken off without any damage to the surface layer and are separately sealed into plastic containers. The plastic containers are marked with the test site name and date of exposure and removal.

4.4 Sulphate Analysis

The volumetric determination of sulphates in the test plates is described below. Any other analytical procedure which gives a satisfactory precision, for example, gravimetric or spectrophotometric methods can also be used.



All dimensions in millimetres.

FIG. 2 EXPOSURE RACK FOR ALKALINE SULPHATION PLATE

4.4.1 *Principle*

The amount of sulphates is determined by the titration of a water/ethanol sample solution with a barium perchlorate water/ethanol titrant in the presence of thorin indicator. The end-point of the titration is indicated by the yellow-pink colour transition in the *p*H interval of 2-3.

4.4.2 *Reagents*

Unless otherwise specified during the analysis reagents of analytical grade and reagent, grade water as per IS 1070 : 1992 shall be used.

4.4.2.1 *Hydrogen peroxide*, solution (150 g/l).

4.4.2.2 *Ethanol*, 95 % (*v*/*v*).

4.4.2.3 *Perchloric acid*, diluted (1 : 5).

4.4.2.4 *Sulphuric acid*, standard solution (0.005 mol/l).

4.4.2.5 *Barium perchlorate*, standard solution (0.005 mol/l).

Dissolve 2.0 g of barium perchlorate trihydrate [$Ba(ClO_4)_2 \cdot 3H_2O$] in water by the addition of perchloric acid (**4.4.2.3**) and adjust it to the *p*H value of 2 to 3 and the solution is diluted to 1 000 ml with ethanol, 95% (*v*/*v*). The exact concentration is determined by the titration of 10 ml of the sulphuric acid standard solution (**4.4.2.4**) following the procedure as described below.

4.4.2.6 *Thorin indicator* (2 g/1) — Dissolve 0.2 g of thorin indicator in 100 ml water.

4.4.3 *Plate Analysis Procedure*

4.4.3.1 *Preparation of the sample solution*

The test plate is taken out from the plastic container and put into an 800-ml beaker and, after wetting, is split into pieces by means of a glass rod. The volume of the suspension is adjusted to 400 ml with water.

1 ml of hydrogen peroxide solution (**4.4.2.1**) is added and the suspension is properly stirred.

The mixture is boiled for a short time and left at rest overnight.

The suspension is vacuum filtered through a quantitative filter paper coarse grade and the residue on the filter is washed twice with water. The volume of the filtrate is adjusted to 500 ml in a volumetric flask (sample solution).

4.4.3.2 *Procedure*

Ten ml of the sample solution is pipetted into a titration flask. With high concentration of sulphates in the sample solution a lower volume may be pipetted and the volume is made up to 10 ml with water. With low concentrations of sulphates in the sample solution 5 ml of sulphuric acid standard solution (**4.2.2.4**) may be added.

Adjust the *p*H to the value between 2 to 3 by addition of perchloric acid (**4.4.2.3**).

40 ml ethanol and 3 drops of thorin indicator solution are added. The solution is titrated by barium perchlorate standard solution (**4.4.2.5**) to the yellow-pink colour transition.

4.5 Expression of Results

The results shall be expressed as sulphur dioxide deposition rate, R, in mg/(m^2.d) according to the formula:

$$R = \frac{m}{A \times t} \qquad \text{... (2)}$$

where

m = total SO_2 content in test plate, in milligrams;

A = area of exposed test plate, in square metres[1]; and

t = exposure time, in days.

For the titration of sulphates by barium perchlorate standard solution, the results are calculated according to the following formula:

$$R = \frac{(V_1 - V_2) \times C_1 \times 64.06 \times V_t}{V_A \times A \times t} \qquad \text{... (3)}$$

where

R = sulphur dioxide deposition rate, in milligrams per square metre day;

V_1 = volume of barium perchlorate standard solution used for titration of the sample, in millilitres;

V_2 = volume of barium perchlorate standard solution used for titration of the blank, in millilitres;

C_1 = concentration of barium perchlorate standard solution in moles per cubic decimetres;

V_t = total volume of sample solution, in millilitres;

V_A = volume of pipetted part of sample solution, in millilitres;

A = area of exposed test plate, in square metres[1]; and

t = exposure time, in days.

5 DETERMINATION OF CHLORIDE DEPOSITION RATE — WET CANDLE METHOD

5.1 Principle

A rain protected wet textile surface with a known area is exposed for a specified time. The amount of chloride deposit is determined by chemical analysis. From the results of this analysis the chloride deposition rate is calculated in $mg/(m^2.d)$.

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[1] A = 0.03 m^2 for the test plate of 150 mm × 100 mm size.

5.2 Sampling Apparatus

5.2.1 *Wet Candle*

The wet candle is formed of a wick inserted into a bottle. The wick comprises a central core of about 25 mm diameter rod of inert material over which is stretched or wound a double layer of tubular surgical gauze or a band of surgical gauze. The surface of the wick exposed to the atmosphere shall be about 100 cm^2, which corresponds to a wick length of about 120 mm. The exposed area shall be accurately known.

One end of the wick is set into a rubber stopper. The stopper has two additional holes through which pass the free ends of the gauze (if tubular gauze is used the lower end is cut along the length of the gauze until about 120 mm is left). The edges of the three holes are shaped as a funnel so that liquid running down the gauze drains down through the stopper (Fig. 3). The free ends of the gauze must be long enough to reach the bottom of the bottle.

The stopper is inserted into the neck of a bottle of polyethylene or any other inert material with a volume of about 500 ml. The bottle containing 200 ml of 20 percent glycerol-water solution added with octanoic acid to prevent the formation of fungus such as *Aspergillus niger*. The solution is made up by mixing 200 ml glycerol $CHOH(CH_2OH)_2$ with distilled water to a volume of 1 000 ml. Add 20 drops of octanoic acid ($C_8H_{16}O_2$) to the solution.

In situations of prolonged extreme temperatures, that is, greater or less than ± 25°C it may be necessary to increase the glycerol content to 40 percent to prevent freezing or excessive evaporation, or to substitute ethylene glycol of 20 percent strength for the glycerol solution.

5.2.2 *Exposure Rack*

The wet candle is exposed on a rack under the centre of a roof as shown in Fig. 4. The roof should be 500 mm × 500 mm, inert and opaque. The candle should be attached so that the distance from the roof to the top of the wick is 200 mm and it is central relative to the roof. The distance between the bottle and the ground level should be at least 1 metre. The candle should have an open access to the sea or other chloride source.

5.3 Sampling Procedure

At the test location the prefabricated candle is installed and controlled in the following steps:

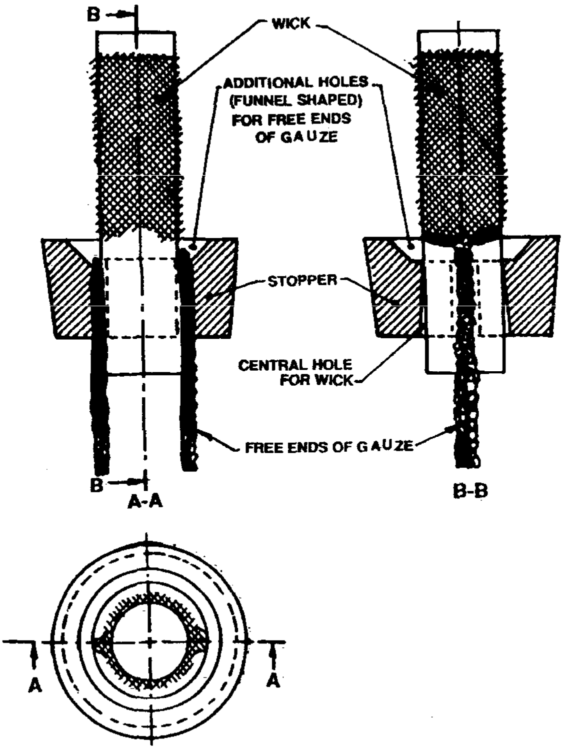
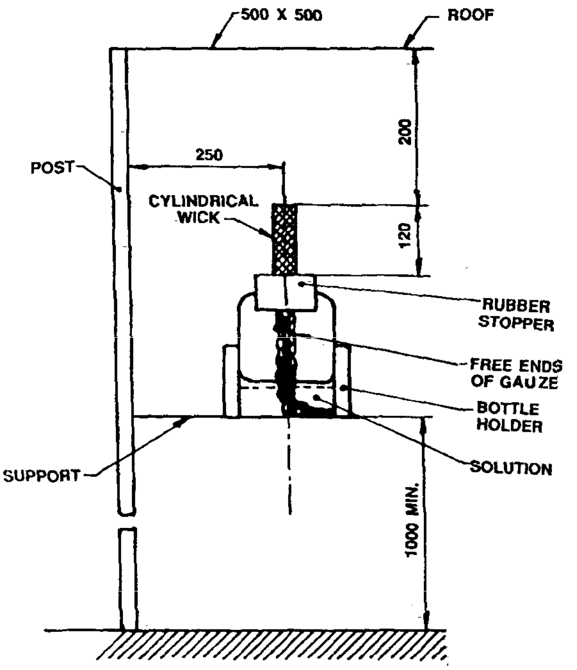


FIG. 3 DETAILS OF RUBBER STOPPER

- Adjust the length of the exposed part of the wick to the desired value
- Remove stopper and wick from bottle, wash free ends of gauze and bottle with distilled water
- Place 200 ml of glycerol-water solution in the bottle
- Reassemble the wick and the bottle
- Place the candle in exposed position as given in Fig. 4.

The glycerol-water solution should be changed at monthly intervals as follows:

- Loosen stopper in bottle.
- Wash down the wick with at least 200 ml of distilled water, carefully, ensuring that the washings run through the drain holes in the stopper and then into the bottle.
- Remove stopper and wick from bottle and wash free ends of gauze as they are withdrawn from bottle.
- Place stopper and wick into new bottle containing 200 ml of fresh glycerol-water solution, ensuring that the free ends of the gauze dip well into the solution. Replace the candle in its holder.
- Screw the lid tightly on the bottle that has been removed, making sure that it does not leak. Mark the bottle clearly with test site, name, location, date exposed and removed.



All dimensions in millimetres.

FIG. 4 SAMPLING APPARATUS ASSEMBLY

5.4 Chloride Analysis

A suitable chloride analysis procedure is described below. Any other analytical procedure which can give a satisfactory result can also be used.

5.4.1 *Principle*

The amount of chloride in a sample is determined by mercurimetric titration in the presence of the diphenyl-carbazone/bromophenol blue mixed indicator. The end point of the titration is indicated by the formation of a blue-violet complex of mercuric diphenyl-carbazone in the *p*H interval of 2.3 to 2.8.

5.4.2 *Reagents*

Unless otherwise specified, during the analysis reagents of recognized analytical grade and reagent grade water as per IS 1070 : 1992 shall be used.

5.4.2.1 *Ethanol,* 95 % *(v/v).*

5.4.2.2 *Nitric acid (0.05 mol/l)* — Dilute 3 ml nitric acid (r.d. = 1.40) to 1 000 ml.

5.4.2.3 *Sodium hydroxide solution (0.25 mol/l)*— Dissolve 10 g sodium hydroxide in water and dilute to 1 000 ml.

5.4.2.4 *Sodium chloride, standard solution (0.025 mol/1)* — Dry the sodium chloride for 1 h at 300°C. Dissolve 1·461 3 g in water and dilute to 1 000 ml in a volumetric flask.

5.4.2.5 *Mercuric nitrate,* standard solution (0.012 5 mol/l).

Dissolve 4.283 0 g of $Hg(NO_3)_2 H_2O$ in 50 ml water acidified with 0.5 ml nitric acid (HNO_3) (r.d. = 1.40). Dilute to 1 000 ml in volumetric flask. Filter if necessary and determine the exact concentration by titration against sodium chloride standard solution as described below.

5.4.2.6 *Mercuric nitrate,* standard solution (0.007 05 mol/1).

Dissolve 2.420 0 g of mercuric nitrate [$Hg(NO_3)_2.H_2O$] in 25 ml water acidified with 0.25 ml nitric acid (r.d. =1.40). Dilute to 1 000 ml in volumetric flask. Filter, if necessary, and determine the exact concentration by titrating against sodium chloride standard solution as described below.

5.4.2.7 *Mixed indicator solution*

Dissolve 0.5 g of diphenyl carbazone and 0.5 g bromophenol blue in 75 ml 95 percent ethanol and dilute to 100 ml with ethanol and keep in a dark bottle in a refrigerator.

5.4.3 *Procedure*

Sample a known part of the chloride sample. This sampled part shall not contain more than 20 mg chloride ions.

If sampled part of the chloride sample contains more than 2.5 mg chloride ions the titration shall be performed using mercuric nitrate [$Hg(NO_3)_2$] solution as specified in **5.4.2.5**.

If the sampled part of the chloride sample contains less than 2.5 mg of chloride ions the titration shall be performed using mercuric nitrate [$Hg(NO_3)_2$] solution as specified in **5.4.2.6** and a microburette. In this case also a blank titration on 50 ml water shall be performed. If the sampled part contains less than 0.1 mg chloride per litre a suitable volume is evaporated to 50 ml.

Add 5-10 drops of mixed indicator.

If a blue-violet or red colour develops add nitric acid (**5.4.2.2**) drop by drop until the colour changes to yellow and then add 1 ml excess of the nitric acid. If a yellow or orange colour forms immediately on the addition of the indicator, develop the blue-violet colour by adding sodium hydroxide solution (**5.4.2.3**) drop by drop then proceed with the acidification.

Titrate the yellow acidified solution with relevant mercuric nitrate solution (**5.4.2.5** or **5.4.2.6**) until a blue-violet colour persists throughout the solution.

5.5 Evaluation of Test Results

The results shall be expressed as deposition rate of chloride, *R* in mg $(m^2.d)$ and are calculated according to the following formula:

$$R = \frac{m}{A \times t} \qquad \dots (4)$$

where

m = total chloride content, in milligrams;

t = exposure time, in days; and

A = area of the exposed gauze surface, in square metres.

If the mercurimetric titration method is used, the deposition rate of Cl^-, R, in mg/(cm^2.d) is calculated according to:

$$R(C1) = \frac{2(V_3 - V_4) \times C_2 \times 35.5 \times V_T}{V_A \times A \times t} \qquad \dots (5)$$

where

V_3 = volume of mercuric nitrate solution used for titration of the sample, in millilitres;

V_4 = volume of mercuric nitrate solution used for titration of the blank, in millilitres;

C_2 = concentration of mercuric nitrate solution, in moles per cubic decimetres;

V_T = total chloride sample volume, in millilitres;

V_A = volume of pipetted part of sample solution, in millilitres;

A = area of the exposed gauze surface, in square metres; and

t = exposure time, in days.

NOTE — The area of the exposed gauze surface can be calculated by multiplying the diameter of the rod by π (3.14) and by the length of the wick protruding from the stopper.

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'.

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